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POLYMER-FILM COATING OF MAGNESIUM FOR PARABOLOIDAL MIRRORS

by Thaddeus S. Mroz and Robert B. King

Lewis Research Center

Cleveland, Ohio

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ABSTRACT

A material and a method were determined for coating large mechanically polished magnesium surfaces to provide a high-quality surface, acceptable for further coating with vapor-deposited optical films. The coating material was a modified epoxy-resin mixture. The application procedure consisted of surface preparation using commercially available chemical cleaning agents, spray application with commercially available equipment, and a carefully programmed curing schedule.

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SUMMARY

An empirical investigation was conducted to establish a method, a material, and equipment requirements for coating of large magnesium surfaces with an epoxy film to a controlled film thickness of $1\frac{1}{2}$ to 2 mils (4×10^{-5} to 5×10^{-5} m). The requirements of major interest were excellent bonding between the magnesium and epoxy and a smooth and uniform film surface that would be a suitable base for subsequent specular coatings.

The mechanically prepolished magnesium surface was chemically treated. An epoxy-resin base coating (Eccocoat type EP-3) was prepared and sprayed onto the treated magnesium surface in a controlled environment. The film was then cured according to a prescribed temperature schedule.

The epoxy-coated magnesium exhibited the following characteristics:

- (1) The magnesium-epoxy bond and coverage were satisfactory.
- (2) The smoothness of the epoxy surface was considered satisfactory.
- (3) The adherence of optical films to the epoxy was satisfactory.

INTRODUCTION

A program was initiated to design and fabricate a one-piece, rigid, 20-foot- (6.1-m-) diameter, flight-type solar concentrator made of magnesium. The objective of the program was to achieve a highly specular reflective surface on the magnesium concentrator.

Magnesium is a desirable construction material for aerospace application because of its high strength-to-weight ratio. The physical properties of magnesium and many of its alloys preclude the attainment of mirrorlike surface finishes with mechanical polishing. Rigorous polishing with fine abrasives to eliminate surface irregularities produces surface relieving, which causes an orangepeel effect. Consequently, mechanical polishing of magnesium will not produce a surface with a high specular reflectance. Use of magnesium for aerospace applications that require high reflectance will necessitate the ap-

plication of a thin, smooth, plastic film on the magnesium surface plus the application of vapor-deposited optical films over the smoothing film. Magnesium is chemically active, forming surface films of oxides and carbonates on exposure to the atmosphere. These surface films markedly affect wetting and bonding of protective organic and inorganic films and degrade the reflectance of polished magnesium. Adhesion of applied films to this material is a prime prerequisite for any application.

The purpose of the plastic smoothing film is to cover all the surface irregularities and provide a new glasslike surface over the magnesium which does not reproduce the surface defects. In addition to this, the plastic must bond well with magnesium, have good spray properties, good viscosity control, good flow properties (leveling), usable pot-life, and must be thermally curable to a hard film. The cured epoxy film must have excellent bonding with vapor-deposited aluminum.

An epoxy coating was chosen on the bases of physical and chemical properties **and** ability to provide the smoothing film.

No information on epoxy coating of magnesium could be found in literature or from industrial sources. Procedures for chemical cleaning of magnesium were located but were not specific for epoxy coating applications. Available information indicated that the wetting and bonding characteristics varied with epoxy formulation and manufacture.

Because of the lack of data and process details, the technical problems of coating magnesium with epoxy were solved by investigations in the following areas:

- (1) Magnesium surface preparation
- (2) Coating modification
- (3) Coating mixing technique
- (4) Coating application
- (5) Coating curing schedule
- (6) Coating evaluation

The structural properties of magnesium were not considered in this investigation. Those magnesium properties connected with coating application are considered. The only changes made in the epoxy formulation were those associated with solvent addition (ref. 1) for proper atomization and spray distribution, leveling, and wetting.

Although the work was carried out in a manner which sought to isolate or control certain properties, it quickly became obvious that a general overall latitude involving coating variability, spray-gun operator variability, and other possible factors would have to be tolerated. But, those factors that could be controlled (e. g., mixing time, temperature, etc.) were rigidly adhered to.

SURFACE PREPARATION

The objective in the surface preparation of magnesium was the chemical cleaning of metal and the subsequent formation of a protective film over the magnesium that would

provide good wetting and bonding of the epoxy. Other factors of major interest included the development of a cleaning cycle suitable for processing large magnesium panels and the attainment of a minimum complexity. Chemical cleaning involves the following processes:

- (1) Solvent vapor degreasing - removal of unsaponifiable mineral oils and greases
- (2) Hot-alkaline immersion cleaning - removal of organic and water soluble oils, vegetable and animal greases, and dirt by detergent action and saponification
- (3) Acid immersion cleaning - removal of surface oxide and scale from the metal surface (ref. 2)

In the initial phase of the investigation, development of the acceptable chemical cleaning cycle required the determination of suitable chemical cleaning agents, processing schedule, and the critical areas of the cleaning process. Stepwise testing was performed. This consisted of testing magnesium plates which ranged in size from $2\frac{1}{4}$ inches (0.057 m) by $2\frac{1}{4}$ inches (0.057 m) to 6 inches (0.15 m) by 12 inches (0.30 m). These plates were mechanically polished to a 15 to 40 rms microinch finish, were chemically cleaned, dried, and sprayed with epoxy. Variables in chemical cleaning consisted of different cleaning solutions, changes in cleaning and rinsing sequence, and changes in time and temperature parameters. However, a fixed procedure was used for drying the cleaned magnesium, epoxy preparation, epoxy application, and epoxy curing. A single epoxy formulation was used in this program. In this investigation, the criteria for evaluation and selection of a cleaning cycle were (1) wetting of cleaned magnesium by epoxy, (2) pitting, and (3) suitable time cycle for use with large panels.

The following cleaning agents were used:

- (1) Solvent cleaning: Fully stabilized missile grade trichloroethylene
- (2) Alkaline cleaning solutions:
 - (a) Trisodium phosphate - sodium carbonate
 - (b) Micro-Mag D-21
- (3) Acid cleaning solutions:
 - (a) Chromic acid and calcium nitrate
 - (b) Chromic acid and sodium nitrate
 - (c) Sulfuric acid
 - (d) Sodium dichromate and nitric acid

The cleaning process was varied to establish the need for any or all of the solvent, alkaline, and acid cleaning steps. The following sequence was investigated:

- (1) Solvent cleaning only
- (2) Alkaline cleaning only
- (3) Acid cleaning only
- (4) Solvent and acid cleaning
- (5) Solvent and alkaline cleaning
- (6) Alkaline and acid cleaning
- (7) Solvent, alkaline, and acid cleaning

Solvent cleaning was performed at 180° F (355 K). Alkaline cleaning was performed over the temperature range of 155° to 165° F (342 to 347 K) followed by an 8-minute hot-water and a 5-minute cold-water immersion rinse. The acid cleaning was performed at room temperature and was followed by a 10-minute cold-water immersion rinse. The wet plates were dried in a clean environment at 135° to 140° F (331 to 333 K). The results of the cleaning tests are shown in table I. The results of the testing indicated that good wetting resulted only when the solvent, alkaline, and acid cleaning steps were performed consecutively.

The following solutions were eliminated for the reasons stated:

- (1) The sulfuric acid solution and the dichromate - nitric acid mixture caused pitting within 10 seconds of immersion.
- (2) The chromic acid - calcium nitrate mixture caused poor wetting.
- (3) The trisodium phosphate - sodium carbonate alkaline cleaner was prohibited from use because of the local water pollution regulations controlling phosphate disposal in sewers.

This testing program established a cleaning cycle which provided good epoxy wetting and bonding characteristics. This cycle is detailed in the appendix.

The following are critical factors in the cleaning cycle:

- (1) The pH of the alkaline and acid solutions must be maintained in the specified range.
- (2) Operating temperatures of the alkaline and acid solutions must be maintained in the specified range to effect good cleaning and avoid pitting in the acid bath.
- (3) Rinsing is extremely important and must be complete. Fresh rinse water must be used.
- (4) The chemically cleaned magnesium must be protected from contamination after cleaning.

COATING MODIFICATION

An epoxy-resin-based coating was selected that did not contain any reactive modifiers. The intention was to use a coating with the maximum amount of cross-linking in order to prevent degradation of the physical or chemical properties (refs. 3 and 4). Three epoxy-based coatings were considered. Two coatings were rejected because they contained proprietary diluents. The coating selected was an available material designated as Eccocoat EP-3.

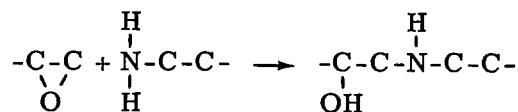
The selected coating consisted of an epoxy resin and an aliphatic amine hardener, which were mixed just prior to use. The characteristics of the sprayed film are determined by the coating formulation and by the resin-hardener reaction rate. Organic sol-

vents were added to reduce the viscosity (ref. 5), improve covering and leveling, and delay the resin-hardener reaction. These solvents were aromatic hydrocarbons, alcohols, and ketones. Aromatic hydrocarbons such as toluene and xylene by themselves did not release readily from the film and did not contribute to good wetting. Although alcohols are at best only latent solvents and probably function as diluents (ref. 6), certain ether-alcohols contributed to good spray properties, as well as good wetting, but were difficult to remove completely at room temperature and resulted in bubbles or pinholes when the coating was heat cured. Ketones were found to be good viscosity reducers, provided good wetting, and could be readily removed from the film. Although acetone, methyl ethyl ketone, xylene, and ether-alcohols were effective, a mixture of 60 weight percent methyl isobutyl ketone and 40 weight percent toluene gave the best overall results and was used on the program.

An acceptable coating was obtained by adding 5 weight percent solvent to 95 weight percent Eccocoat EP-3.

COATING MIXING TECHNIQUE

An epoxy resin cures or hardens by a chemical reaction between the epoxy groups in the resin and some other group provided by the hardening agent used (refs. 3 and 4). A typical example using an amine hardener illustrates the process:



Note that the remaining hydrogen on the amine group can react with another epoxy group to form a three-dimensional, cross-linked structure. The reaction is quite slow at room temperature, but since it is exothermic the rate can increase with time, depending on the geometric shape of the mass. A compact mass, such as a cylinder, conserves the exothermic heat with a resulting increase in reaction rate. The temperature rise is rapid, causing the reaction to become violent and resulting in foaming, bubbling, and a general decomposition of the coating. The desirable technique is to mix the components to initiate the reaction and then apply them as a thin film, which hardens or solidifies slowly at room temperature. This mixture will not react completely under these conditions, and a subsequent exposure to heat is required. The slow curing at room temperature allows the leveling of the film to proceed and the volatile constituents to escape so that they will not cause bubbles or pinholes when heat is applied.

The resin and hardener should be mixed so that there are no localized high concen-

trations of hardener at any time. These high concentrations can cause improper chemical linkages and can produce small globules that create undesirable discontinuities in the coating. The slow addition of hardener to a well-stirred resin avoids these problems.

The solvent composition (ref. 1) influences the curing reaction in at least four ways: (1) dilution of the resin and/or hardener concentration contributes to a slow and uniform inception of the resin-hardener reaction; (2) the rate of solvent evaporation establishes the coating temperature at the coating-air interface; (3) moisture compatibility of the solvent prevents degradation of the surface and curing properties (if the surface temperature falls below the dew point of the surrounding atmosphere, water will condense on the surface and impede curing); (4) functional groups such as carbonyls and ketones appear to decrease the rate of resin hardening, thereby allowing more time for the application of the coating before it becomes too viscous for use.

The best results were obtained by slowly pouring the hardener into the stirred epoxy resin. Good agitation by means of a standard air-powered propeller-type mixer was maintained for 12 minutes to age the coating. The mixed coating material was then poured immediately into the spray equipment and applied to the magnesium substrate. This procedure was carefully followed.

COATING APPLICATION

The mixed and aged coating was applied with a commercially available spray gun equipped with a pressure cup. By using a fan spray and with the spray gun held 6 to 8 inches (0.15 to 0.20 m) from the magnesium, a film was applied with 5 psi (3.5×10^4 N/m²) cup pressure and 45 psi (3.2×10^5 N/m²) atomizing pressure. Excessive atomizing pressure was avoided to prevent blowing or otherwise degrading the film. Dry filtered nitrogen gas was used to prevent introduction of water or other contaminants. Very thin films were laid down - just enough to give a continuous liquid layer on each pass of the spray gun. Three passes were required to obtain the desired thickness of $1\frac{1}{2}$ to 2 mils (4×10^{-5} to 5×10^{-5} m), which was sufficient to cover the surface irregularities.

The application of individual thin films provides excellent conditions for solvent removal because it prevents pinholes or bubbles during the heat cure, and it increases the viscosity of the applied film so that the film will not creep, crawl, drape, or run (ref. 7).

Subsequent passes deposit more low-viscosity material which flows out readily. The solvent in the freshly applied material mixes with the previously applied layer. At the same time, solvent evaporates from the surface. Both of these processes allow for good leveling of the epoxy over a very short period of time (2 to 10 sec) to obtain a smooth, homogeneous film, while a higher viscosity, which resists movement (creep, crawl, etc.), is quickly produced. Subsequent passes of the spray gun continue this action to build up a

desirable thickness. The total coating thickness must be thin enough to allow the solvent to escape in a reasonable time at room temperature. This must occur before the resin-hardener reaction hardens the film. We found that 60 minutes at room temperature was generally sufficient for adequate solvent removal. Other considerations require that the coating be thick enough so that sufficient film integrity is achieved to provide adequate mechanical strength as well as to prevent transport of water or other material through the film which might cause its degradation. We found that a film thickness of $1\frac{1}{2}$ to 2 mils (4×10^{-5} to 5×10^{-5} m) met all the requirements.

A successful coating application was contingent on a skillful and experienced spray-gun operator and strict control of cleanliness. Foreign particulate matter or contamination of a cleaned surface resulted in coating defects. Typical coating defects that were experienced are illustrated in figure 1.

CURING SCHEDULE

As noted earlier, the epoxy resin-hardener reaction will not go to completion in the desired manner in any reasonable time without the application of heat. The curing cycle starts the moment the resin and hardener are mixed. There is a period after mixing during which the rate of the curing reaction increases slowly. This induction period is a function of temperature, the manner in which the resin and hardener are mixed, and the solvent composition of the mixture.

It was found by trial and error that a 12-minute continuous mixing after the addition of the hardener gave the best mixture for spraying. This resin-hardener mixture is aged for 15 minutes before application. The sprayed coating is allowed to stand at room temperature for 60 minutes to allow most of the solvents to evaporate. It is then cured according to the detailed schedule shown in table II. This schedule, which provides for slow incremental temperature increases, completes the solvent removal process before the film is totally cured. It further causes progressive curing, which increases the viscosity of the coating and prevents running or creeping as the temperature is raised. A curing assembly, utilizing infrared lamps, is shown in figure 2.

EVALUATION OF COATINGS

The cured epoxy films were tested for (1) required thickness of plastic film to cover the magnesium surface irregularities, (2) adherence of the epoxy film to the magnesium surface, (3) quality of the epoxy surface, and (4) adherence of optical films to the epoxy surface.

The tests produced the following results:

Coverage. - An epoxy film thickness of $1\frac{1}{2}$ to 2 mils (4×10^{-5} to 5×10^{-5} m) could cover the surface irregularities associated with an 8 to 20 rms microinch magnesium surface finish.

Epoxy-magnesium bonding. - The epoxy-magnesium bond successfully withstood 20 instantaneous thermal shocks from -320° to 212° F (78 to 373 K). The bond also successfully withstood a 2-month exposure to a reduced pressure of 1×10^{-6} millimeter of mercury (1.33×10^{-4} N/m²).

Quality of epoxy surface. - The smoothness of the epoxy surface was considered satisfactory. A reflectance measurement of the epoxy surface which was coated with vapor-deposited aluminum showed a specular reflectance of 88 percent.

Adherence of optical films. - Aluminum and silicon monoxide vapor-deposited films were successfully applied to the epoxy surface. Tape tests and exposure of the films to a temperature of -320° F (78 K) showed good adherence of the films to the epoxy surface.

CONCLUDING REMARKS

A method was developed for applying a commercially available epoxy-resin-based coating to a magnesium substrate to be used as a solar collector. The success of this method was contingent on the adherence to detail in the surface preparation, coating formulation, mixing technique, spray technique, and curing schedule. Testing with an aluminum reflecting surface indicated that acceptable mechanical and optical properties were obtained.

Potential sources of trouble were avoided wherever possible. The coating-chamber environment was meticulously controlled. Dust in the air was filtered out by 5-micron absolute filters. Relative humidity was maintained at 60 percent and the temperature was maintained at 72° F (295 K). Presence of moisture or oil in the atomizing gas was eliminated through the use of dry nitrogen. Rigid observance of these requirements was found necessary for acceptable coatings.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, May 29, 1968,

120-33-06-04-22.

APPENDIX - PROCEDURE FOR CHEMICAL CLEANING OF MAGNESIUM

The chemical cleaning process is performed in the following sequence by using the stated system parameters.

- (1) Solvent cleaning. Immersion in vapors of fully stabilized trichloroethylene for a period of 8 to 10 minutes followed by a spray rinse with hot trichloroethylene
- (2) Hot alkaline cleaning. Immersion in a hot alkaline circulating bath consisting of Micro Mag D-21 water solution for a period of 8 to 10 minutes with a solution pH of 11.5 to 13.0 at a temperature of 155⁰ to 165⁰ F (342 to 347 K)
- (3) Water rinse
 - (a) Hot water spray rinse for a period of 8 to 10 minutes at a water temperature of 155⁰ F (342 K)
 - (b) Cold water spray rinse for a period of 5 minutes with a water temperature of 60⁰ to 70⁰ F (289 to 294 K)
- (4) Acid cleaning. Immersion in a circulating solution of chromic acid and sodium nitrate for a period of 7 to 9 minutes with a solution pH of 0.5 to 1.0 and a maximum solution temperature of 70⁰ F (294 K)
- (5) Water rinse. Cold water spray rinsing for a period of 10 minutes with a water temperature of 60⁰ to 70⁰ F (289 to 294 K)
- (6) Drying of cleaned panel. Infrared lamp drying for a period of 1 hour at 135⁰ to 140⁰ F (331 to 333 K)

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TABLE I. - EXPERIMENTAL CLEANING PROCESSES

Test sequence	Cleaning process						Results
	Cleaning agent	Concentration		Temperature		Immersion time	
		oz/gal	kg/m ³	°F	K		
Solvent cleaning							
1	Stabilized trichloroethylene	---	---	180	355	5 to 6 min	Poor wetting
Alkaline cleaning							
2a	Trisodium phosphate and sodium carbonate	4 4	30 30	155 to 165	342 to 347	8 min	Poor wetting
b	Micro-Mag D-21	8	60	155 to 165	342 to 347	8 min	Poor wetting
Acid cleaning							
3a	Chromic acid and sodium nitrate	24 4	180 30	70	294	8 min	Poor wetting
b	Chromic acid and calcium nitrate	20 1.5	150 11	70	294	8 min	Poor wetting
c	Sodium dichromate and nitric acid	24 16	180 120	70	294	8 sec	Poor wetting
d	Sodium dichromate and nitric acid, followed by chromic acid and sodium nitrate	24 23 24 4	180 172 180 30	70	294	8 sec 6 min	Poor wetting
e	Sulfuric acid	9	65	70	294	5 sec	Poor wetting
Solvent and alkaline cleaning							
4a	Sequence 1 followed by 2a						Poor wetting
b	Sequence 1 followed by 2b						Poor wetting
Solvent and acid cleaning							
5a	Sequence 1 followed by 3a						Poor wetting
b	Sequence 1 followed by 3b						Poor wetting
c	Sequence 1 followed by 3c						Poor wetting
d	Sequence 1 followed by 3d						Poor wetting
e	Sequence 1 followed by 3e						Poor wetting
Alkaline and acid cleaning							
6a	Sequence 2a followed by 3a						Poor wetting
b	Sequence 2a followed by 3b						Poor wetting
c	Sequence 2a followed by 3c						Poor wetting
d	Sequence 2a followed by 3d						Poor wetting
e	Sequence 2a followed by 3e						Poor wetting
Solvent, alkaline, and acid cleaning							
7a	Sequence 1 followed by 2a followed by 3a						Good wetting
b	Sequence 1 followed by 2a followed by 3b						Spotty wetting
c	Sequence 1 followed by 2a followed by 3c						Good wetting
d	Sequence 1 followed by 2a followed by 3d						Good wetting, with slight pitting
e	Sequence 1 followed by 2a followed by 3e						Good wetting, with slight pitting
Selected cleaning cycle (solvent, alkaline, and acid)							
8	Sequence 1 followed by 2b followed by 3a						Good wetting

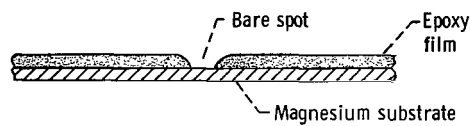
TABLE II. - CURING SCHEDULE

[Curing was performed in a controlled environment; the energy output of the infrared iodine-quartz curing lamps was directed to the noncoated side of the magnesium.]

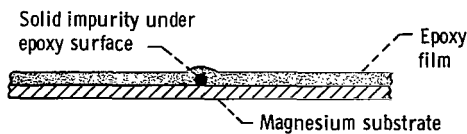
Curing temperature ^a		Curing time, hr
°F	K	
^b 72	295	1
85	303	1
95	308	1/2
105	314	1/2
125	325	1/2
140	333	1/2
155	342	1
175	353	1
185	358	2

^aCuring temperatures represent temperature of magnesium.

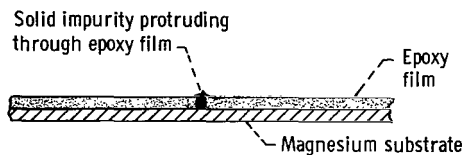
^bRoom temperature.



(a) Nonwetting.



(b) Foreign particle in film under surface of epoxy.



(c) Foreign particle in film protruding through surface of epoxy.

Figure 1. - Typical coating defects.

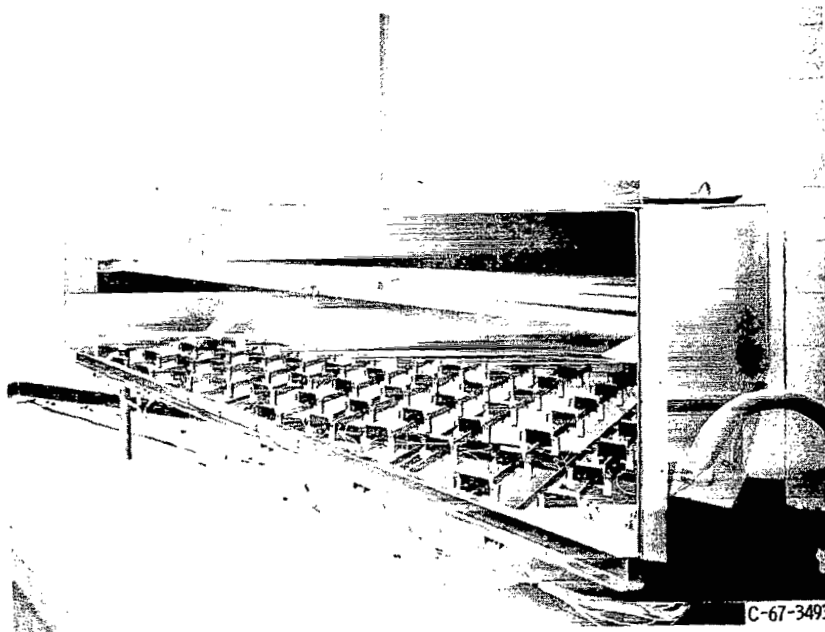


Figure 2. - Curing equipment.

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